1/15/79

STATUS REPORT: RECLAIMED FIBER UTILIZATION

Project 2697-3--Optimization of Recycled Fiber in Linerboard

Table of Contents

I. Introduction

The first part of the project is concerned with the addition of composite old corrugated to linerboard. The following phases of the work were completed or were in progress during 1978.

1. Procurement of equipment and fiber supplies (see January 1978 status report)

2. Characterization of fiber supplies (see January and April 1978 status reports)

3. Effect of distribution of OCC between primary and secondary furnishes (see April 1978 status report)

4. Effect of amount of OCC (see July 1978 status report)

5. Effect of ozonation on recycled fiber properties (see July and October 1978 status reports; Progress Report One, Project 2697-53, September 24, 1978. Additional work in process is summarized herein).

6. Chemical treatments and additives (research in process on chemical treatments other than ozonation is summarized in this report).

7. "Control" beater curves on OCC and virgin kraft furnishes at standard sheet weight and two wet pressing pressures. In order to provide appropriate bench-marks to assess the effects of ozonation and other chemical treatments/additives, beater curves have been obtained on the OCC and virgin kraft furnishes at two wet pressures and the results are summarized herein.

8. Refining variables and techniques (see July and October 1978 status reports. Further work is being planned but is not discussed in this report).

II. Budget

The cost and time schedules in Part IX of the research plan dated August 25, 1978, have been revised to reflect the proposed 1979 FKBG budget of $40,000 and the corresponding decrease in IPC funding to $40,000. This results in a total loss in project funding of $40,000 per year -- $20,000 from FKBG and $20,000 from the Institute. The time schedules project the lower budgeting rates by FKBG and IPC into the future. It is now estimated that the project completion will be delayed by about one year, i.e., from late 1980 to late 1981.

Copies of the revised schedules and research plan are included in the meeting material. The schedule changes are paged from 72 to 77 and replace the schedules shown in the August 25, 1978 revision of the research plan which was forwarded to you on October 20, 1978.

To clarify the project funding, the FKBG and IPC manpower and funding are tabulated separately in the Project Summary Schedule Sheet (Figure 1). The different fiscal years (FKBG - January 1-December 31 and IPC - July 1-June 30) should be kept in mind as they cause minor differences between FKBG and IPC funding.

During 1978, research on the project indicated that ozone treatments significantly increase most strength properties without any major reduction in freeness. The process will present little or no pollution problem and have no detrimental effect on the white water system. Ozonation operating costs to obtain a 35% burst improvement are estimated to range from about $12-$14 per ton of treated fiber. When capital requirements are included,

			1977		1978		1979		1980		1981	
III--COMPOSITE RECYCLED OCC (FKBG)												
IV--FRACTIONATED OCC (FKBG)												
V--FUNDED PULP TREATMENTS (IPC)												
VI--ASPHALT DISPERSION PROCESS (FKBG)												
VII--SELECTION OF CANDIDATE APPROACHES (FKBG)												
VIII--MACHINE TRIALS (FKBG)												
FKBG--PARTS III, IV, VI, VII, VIII												
MANPOWER (MAN-YR)				0.5	0.4	0.6	0.3	0.4	0.3	0.4	0.3	0.4
CASH FLOW--EQUIP. ($000)				15	15	--	5		5	--	5	--
--TOTAL ($000)				40	30	30	20	20	20	20	20	20
CUMULATIVE TOTAL ($000)				40	70	100	120	140	160	180	200	220
IPC--PART V												
MANPOWER (MAN-YR)				0.8	0.7	0.5	0.5	0.4	0.3	0.3	0.4	0.2
CASH FLOW--EQUIP. ($000)				--	--	5	--	--	5	5	--	--
--TOTAL ($000)				40	40	30	30	20	20	20	20	10
CUMULATIVE TOTAL				40	80	110	140	160	180	200	220	230
COMBINED												
MANPOWER (MAN-YR)				1.3	1.1	1.1	0.8	0.7	0.6	0.7	0.7	0.6
CASH FLOW--CUMULATIVE TOTAL				80	150	210	260	300	340	380	420	450

Figure 1. Project Schedule Summary Sheet

the total costs are estimated to range from \$16.50 to \$19.50 per ton. If a 35% burst improvement was achieved by increasing the basis weight of commercial linerboard, the cost is estimated to range from \$43-\$64 per ton of linerboard. Thus the ozonation process has advantages in the treatment of pulp fibers to upgrade performance without sacrificing productivity.

At the \$60,000 budget rate and matching IPC funding (total of \$120,000/year) we anticipated that the commercial potential of the ozonation process relative to other chemical treatments/additives would be completed in 1979. The proposed lower budgeting rate (total of \$80,000 per year) will extend this decision point into mid-1980.

In addition, the present laboratory ozonation equipment and process are far removed from possible pilot and commercial processing. The critical areas are the reactor and, to a lesser extent, the paper fluffing/dewatering of the stock. By mid-1979 the ozonation equipment must be upgraded to simulate commercial processing. Major systems and process analyses will be needed to successfully develop the process to the pilot and commercial stages. At the \$40,000 FKBG and IPC funding rate, these process developments can not be initiated in 1979. Thus, a major loss in the momentum of development of the ozonation process will occur.

In this connection, there is considerable industry interest in oxygen bleaching and pulping and the use of O_2 and O_3 for effluent treatment. Because the ozonation of recycled fiber will require relatively small O_2 capacity, the ozonation process costs will become more favorable for mills already having O_2 generation capacity. Thus, earlier assessment of the commercial feasibility of ozonation will be helpful in mill planning because of the long lead times normally required.

At the current $60,000 budget rate ($120,000 including matching Institute funds), it was anticipated that the research on composite OCC and on fractionated stocks would be substantially completed in 1979. At the $40,000 rate ($80,000 with matching Institute funding), the completion date for these two phases of the program will be delayed until early 1981. The final mill trials to prove out methods of treating recycled fiber will then be delayed until near the end of 1981. Thus, all phases of the program are materially delayed by the budget reduction.

At present, an experienced team of four staff members is being utilized on this project. The team is made up of one technician and three (two essentially full-time and one part-time) professional staff members. Substantial support services are being provided to the program by members of the Chemical Science Division and the Material Sciences Group. Chemical Science Division personnel are providing sheet making assistance and input on chemical treatments. The Material Sciences Group is assisting in handsheet evaluations. In addition, the Institute is seeking a senior level professional man for assignments in the recycled fiber area.

The proposed reduction in the FKBG budget for 1979 from $60,000 to $40,000 and the accompanying reduction in IPC funding to the same level will reduce the manpower assigned by about one-third. This will make it impossible to proceed with the process analysis required for development of the ozonation or alternative processes in 1979. Also, it will lead to inefficiencies in manpower utilization because of personnel scheduling on other assignments and the consequent loss in continuity of the team effort.

Based on the above, we believe that retaining the current $60,000 FKBG budget rate has the following advantages:

1. Promotes more timely evaluation of the commercial feasibility of the ozonation process relative to other treatments of recycled fiber.

2. Fully utilizes the matching Institute funding which otherwise will be reduced ($60,000 IPC vs. $40,000 IPC).

3. Makes better use of manpower presently available for the project.

4. Maintains momentum of the planned work.

5. Promotes earlier utilization of the research.

III. Ozonation of Recycled Fiber

Initial studies reported at the July FKBG meeting and in the subsequent Progress Report entitled Effect of Ozonation on Recycled Fiber Properties (Project 2697-53; September 24, 1978) indicated ozonation will significantly improve the physical properties of OCC. This improvement occurs without significant loss of freeness, fiber length, or apparent attendant pollution problems associated with alternative treatment or processing methods. Therefore, further studies are in progress to define and optimize the variables associated with the ozonation process. In addition, studies are being carried out on commercial OCC to determine if the contaminants remaining after cleaning will affect ozonation. Accordingly, the following series of studies were initiated to advance these goals.

Ozonation of Commercial OCC

Samples of commercial OCC were obtained from Menasha Corporation. Samples were obtained before and after the asphalt dispersion (A/D) process. Upon receipt, the pulp was centrifuged to remove excess water, fluffed, ozonated, formed into handsheets and the physical properties were determined in accordance with procedures established for the IPC model OCC as described in Progress Report One, Project 2697-53. Single trials at ozonation levels of approximately 2.3% and 4.5% ozone consumed based upon the o.d. weight of the fiber were performed.

These O_3 consumptions correspond to treatment times of 15 and 30 minutes. In previous trials on the Institute "model" OCC, treatment times ranging up to 90 minutes were employed. For these comparisons, it was believed that the shorter treatments and lower O_3 consumptions would be of more practical interest.

Table I tabulates the physical test data. Figure 2 shows that the ozone consumptions obtained on the commercial OCC were about the same as obtained on the Institute "model" OCC at equal treatment times. In Figure 2, the O_3 consumptions on the "before" A/D stock are plotted; however, similar results were obtained on the "after" A/D stock. The commercial OCC stocks also exhibited small decreases in freeness with increasing ozonation in the same manner as the Institute furnish used in previous work. Thus the small amounts of contaminants remaining in the commercial OCC did not change the O_3 consumption rates and freeness trends.

Figure 3 indicates that the tensile and bursting strengths obtained on the ozonated commercial OCC stocks exhibit strength increases which are similar to those obtained on the IPC "model" OCC. The pulp obtained after the asphalt dispersion process exhibits lower burst and tensile strengths than the stock obtained after the A/D process as expected from the literature. However, the A/D process does not appear to affect the rate of improvement in burst and tensile with increasing ozonation. The "before" A/D commercial OCC exhibited about the same strength levels as the IPC "model" stock.

Figure 4 shows that the rates of change of the tearing strengths of the commercially cleaned and ozonated OCC were about the same as the ozonated IPC "model" OCC. The tearing strengths of the "before" A/D OCC were lower than the "after" A/D OCC and at the same level as the IPC "model" OCC at all ozonation levels.

TABLE I

PROPERTIES OF HANDSHEETS PREPARED FROM COMMERCIAL OCC
OBTAINED FROM MENASHA CORPORATION

	Before AD Process			After AD Process			FKBG Composite OCC[a]		
Ozonation time, min	0	15	30	0	15	30	0	15	60
Ozone Applied, % of o.d. fiber	--	2.52	4.96	--	2.47	50.2	--	2.35	9.41
Ozone Consumed, % of o.d. fiber	--	2.44	4.66	--	2.29	4.75	--	2.31	8.53
C.S. freeness, cc	550	535	520	590	570	550	633	617	567
% Change	--	-2.7	-5.5	--	-3.4	-6.8	--	-2.5	-10.4
Basis weight, lb/M ft^2	13.8	13.3	13.5	13.2	13.4	13.4	13.2	13.2	13.5
Caliper, points	5.7	5.2	5.2	5.4	5.2	5.1	6.1	5.6	5.3
Apparent density	2.42	2.55	2.57	2.45	2.56	2.63	2.16	2.34	2.54
% Change	--	+5.4	+6.2	--	+4.9	+7.3	--	+8.3	+17.6
Bursting strength, psig	20.5	24.2	29.2	18.2	22.3	24.8	17.2	23.2	33.1
Factor	1.49	1.82	2.17	1.38	1.66	1.86	1.30	1.76	2.45
% Change	--	+22.2	+45.6	--	+20.3	+34.8	--	+35.4	+38.5
Mod. ring compression, lb/inch	4.0	4.1	4.1	3.8	4.0	3.9	3.8	4.0	4.8
Factor	0.290	0.312	0.305	0.285	0.302	0.292	0.284	0.307	0.353
% Change		+7.6	+5.2		+6.0	+2.5	--	+8.1	+24.3
Tear, grams	93.1	85.2	79.2	109.2	99.2	90.0	87.6	85.1	71.9
Factor	6.75	6.41	5.89	8.30	7.39	6.73	6.64	6.45	5.31
% Change	--	-5.0	-12.7	--	-11.0	-18.9	--	-2.9	-20.0
Tensile, lb/inch	13.1	15.6	16.6	11.3	13.5	15.1	12.4	15.6	20.0
Factor	0.95	1.17	1.24	0.86	1.01	1.13	0.94	1.19	1.48
% Change	--	+23.2	+30.5	--	+17.4	+31.4	--	+26.6	+57.4
Stretch, %	2.34	2.60	2.50	2.55	2.90	2.99	1.86	2.18	2.43
% Change	--	+11.1	+6.8	--	+13.7	+17.3	--	+17.2	+30.6
Et, lb/inch	1754	1926	2083	1512	1740	1918	1640	1946	2326
Factor	127.4	144.8	154.9	114.9	129.7	143.4	124.2	147.8	172.0
% Change	--	+13.7	+21.6	--	+12.9	+24.8	--	+19.0	+38.5
TEA, ft-lb/ft^2	2.7	3.6	3.6	2.6	3.5	4.1	2.0	2.9	4.1
% Change	--	+33.3	+33.3	--	+34.6	+57.7	--	+45.0	+105.0

[a] Data obtained from Progress Report 1, Project 2697-53, entitled "Effect of Ozonation on Recycled Fiber", dated September 24, 1978

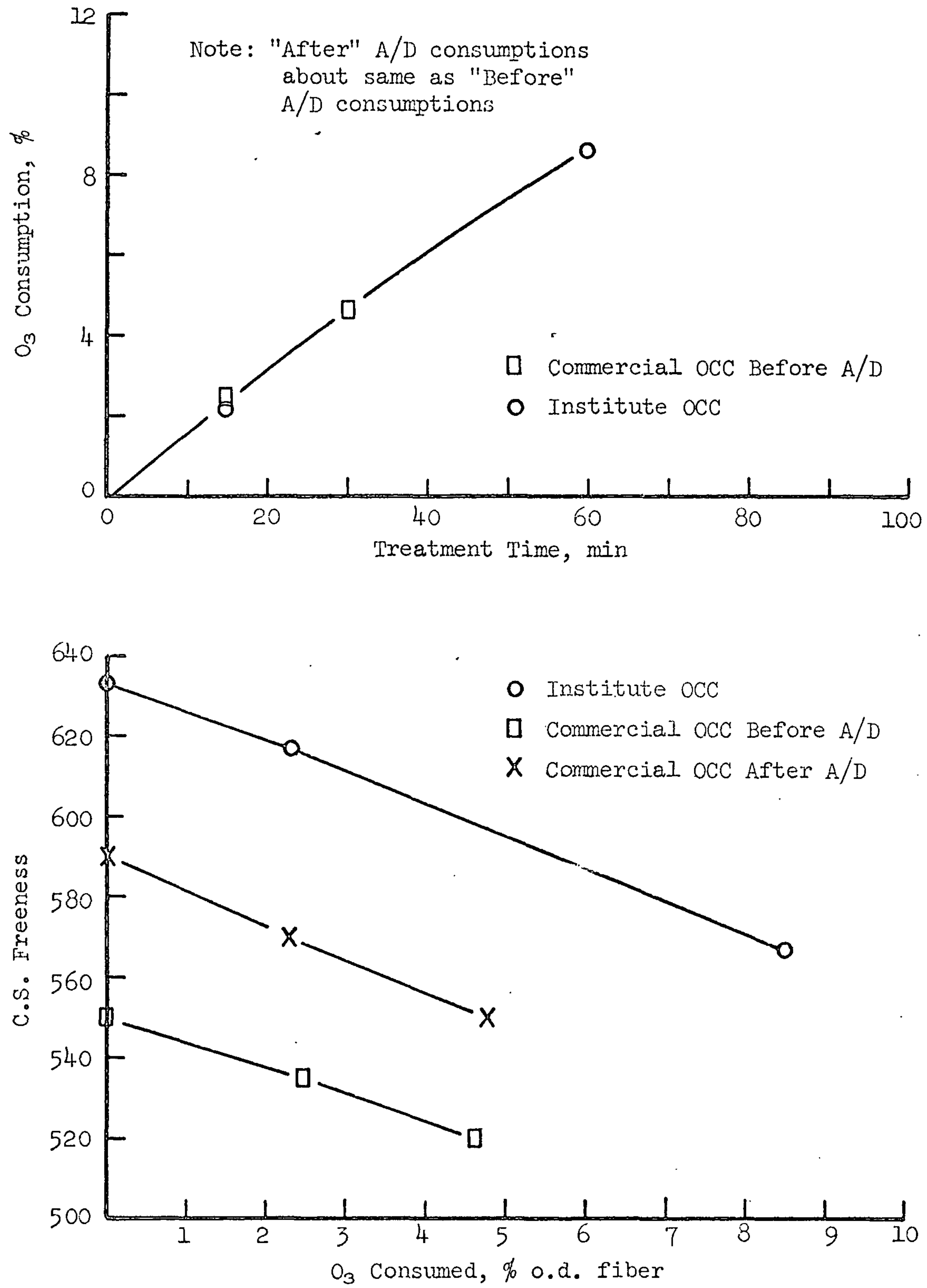

Figure 2. Comparison of Ozone Consumption and Freeness on Commercial OCC and Institute "Model" OCC

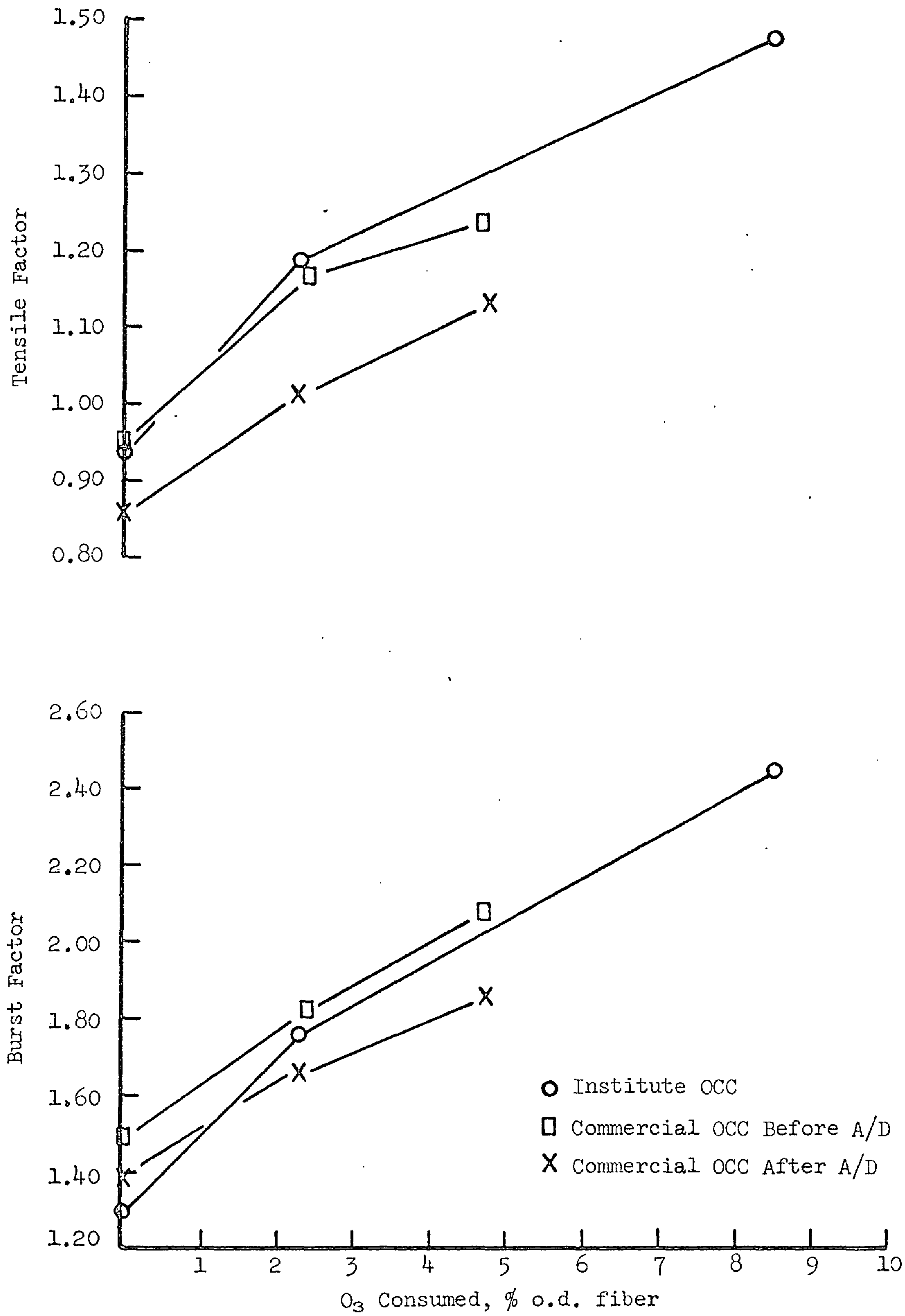

Figure 3. Comparison of Tensile and Burst Factors on Commercial OCC and Institute "Model" OCC

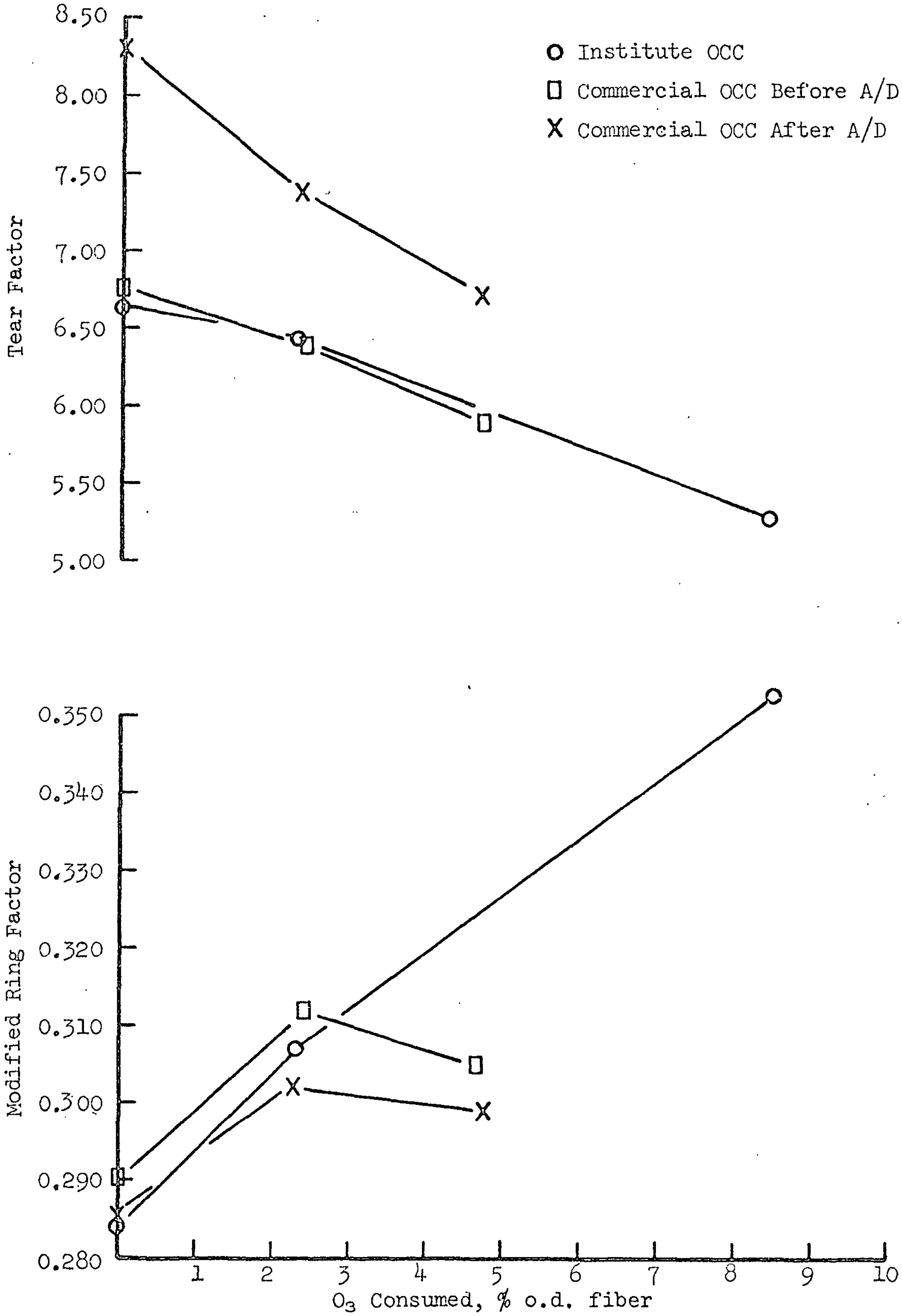

Figure 4. Comparison of Tear and Modified Ring Factors on Commercial OCC and Institute "Model" OCC

The modified ring compression results in Figure 4 show that ozonation increased the strength of the commercial OCC stocks at the 2+% O_3 level in about the same amount as the IPC model OCC. At the 4.5+% O_3 level, the ring strengths of the commercial OCC did not increase in the same way as the IPC stocks; however, compression tests on "thin" sheets are difficult to carry out and tend to be erratic. Review of the tabular data indicated that other physical properties follow the same trends with increasing ozonation as the Institute "model" OCC.

The commercial pulp after application of the asphalt dispersion process exhibits an apparent real loss of strength when compared to the pulp before asphalt dispersion. However, application of the asphalt dispersion process does not appear to affect the rate of strength improvement upon ozonation. Initial properties of the Menasha pulp before asphalt dispersion more closely simulate the strength properties of the IPC model OCC.

Visual examinations of the fibers and handsheets utilizing the SEM (scanning electron microscope) and optical microscopes were performed as a part of the over-all study and generally compared favorably with the IPC model OCC. Several limited differences were noted. The Menasha pulp appeared to present a more "coated" surface upon SEM viewing. Pigment particles also were observed on the fiber surfaces. Limited evidence suggests that they are clay and titanium pigments. As a result, total ash was determined on the samples as well as the IPC model OCC. Results indicate that commercial OCC contains about 2.5% ash as compared with 1.3% for the model OCC. Total solubles and total organic carbon in the solubles are being considered as possible future tests in an effort to provide further characterization of contaminants remaining in commercial OCC. It is expected that significant solubles will be found in the commercial pulps based upon the observations of the water removed during centrifuging.

Fiber analysis indicates a broader array of pulps and species were found in the commercial material, including small quantities of bleached and unbleached softwood and hardwood sulfites and krafts and monocotyledon (probably straw). The major components of the furnish were softwood kraft and hardwood NSSC similar to the IPC model OCC. Ratios of the softwood and hardwood are similar to the IPC model OCC.

Inland Container Corporation, The Chesapeake Corporation of Virginia, and Union Camp Corporation were contacted for further samples to continue these studies, and a sample is on hand from Chesapeake Corporation.

Ozonation of Liner Fractions

In previous studies the OCC composite was ozonated; however, it was hypothesized that ozonation of the long and short fiber fractions separately might be more effective in terms of overall strength improvements at reduced cost. Different pulp furnishes might also respond differently to ozonation. Previous C-stain fiber studies suggested such possibilities could occur. Initial studies with these potentials in mind were therefore undertaken.

The liner and medium fractions of the IPC model OCC were separated by soaking, disintegrated, fluffed, and ozonated in accordance with procedures previously established for the OCC composite. This separation yielded a liner fraction which is essentially 100% softwood kraft and a medium fraction of approximately 80% hardwood NSSC and 20% softwood kraft.

Single trials at ozonation levels of approximately 2.3% and 4.5% ozone consumed based upon the o.d. weight of the pulp were performed. The fractions were formed into handsheets and the usual physical tests were performed.

Physical test results are shown in Table II. Graphs of freeness, tensile, burst, tear and modified ring vs. O_3 consumption are shown in Figures 5-7. Comparable results for the composite are also shown. The trends in percentage strength improvement for the liner and medium are very similar even though initial strength values for the liner and medium are quite divergent. This suggests that both furnishes respond about equally well to ozone treatment. However, it may still be desirable to fractionate and treat only one fraction for economic reasons and/or avoid further fiber shortening and reduction of freeness. This would be particularly true for the hardwood furnish. Properties of the OCC composite generally are closer to the properties of the liner fraction as might be expected since approximately 70% of the composite is softwood kraft.

C-stain and SEM studies were undertaken. C-stain color shift trends with increasing ozonation were similar to those reported for the various softwood and hardwood components of the composite OCC, although responses to color shift to the blue appeared to occur at a slightly faster rate than for the comparable composite.

SEM studies exhibited some differences. The trend of "erosion" and fibrillation on the liner (softwood) fraction could be observed. However, these same trends were not readily observed on the medium (hardwood) fraction. Previous C-stain studies on the composite have shown that

TABLE II

COMPARISON OF PROPERTIES OF HANDSHEETS PREPARED FROM OZONATED LINER AND MEDIUM FRACTIONS WITH COMPOSITE OCC

	Liner			Medium			Composite OCC[a]		
Ozonation time, min	0	15	30	0	15	30	0	15	60
Ozone applied, % of o.d. fiber	--	2.43	4.79	--	2.46	4.77	--	2.35	9.41
Ozone consumed, % of o.d. fiber	--	2.35	4.48	--	2.34	4.41	--	2.31	8.53
Reaction efficiency, %	--	96.7	93.5	--	95.1	92.5	--	98.3	90.7
C.S. freeness, cc	723	678	670	395	375	380	633	617	567
% Change	--	-6.2	-7.3	--	-5.1	-3.8	--	-2.5	-10.4
Basis weight, lb/M ft^2	13.6	13.3	13.4	13.4	13.5	12.8	13.2	13.2	13.5
Caliper, points	5.6	5.2	5.0	6.3	5.8	5.4	6.1	5.6	5.3
Apparent density	2.44	2.55	2.65	2.14	2.35	2.37	2.16	2.34	2.54
% Change	--	+4.5	+8.6	--	+9.8	+10.7	--	+8.3	+17.6
Bursting strength, psig	17.9	23.7	26.2	13.7	19.6	21.1	17.2	23.2	33.1
Factor	1.32	1.79	1.96	1.02	1.46	1.65	1.30	1.76	2.45
% Change	--	+35.6	+48.5	--	+43.1	+61.8	--	+35.4	+88.5
Mod. ring compression, lb/inch	3.8	4.1	4.5	4.3	4.8	4.6	3.8	4.0	4.8
Factor	0.278	0.309	0.336	0.322	0.358	0.359	0.284	0.307	0.353
% Change	--	+11.2	+20.9	--	+11.2	+11.5	--	+8.1	+24.3
Tear, grams	116.4	103.0	98.8	38.6	40.0	37.6	87.6	85.1	71.9
Factor	8.59	7.75	7.38	2.89	2.97	2.94	6.64	6.45	5.31
% Change	--	-9.8	-14.1	--	+2.8	+1.7	--	-2.9	-20.0
Tensile, lb/inch	12.2	14.8	16.2	12.2	15.2	15.1	12.4	15.6	20.0
Factor	0.90	1.12	1.21	0.91	1.13	1.18	0.94	1.19	1.48
% Change	--	+24.4	+34.4	--	+24.2	+29.7	--	+26.6	+57.4
Stretch, %	2.08	2.32	2.26	1.70	2.03	2.14	1.86	2.18	2.43
% Change	--	+11.5	+8.7	--	+19.4	+25.9	--	+17.2	+30.6
Et, lb/inch	1646	1899	2113	1675	1967	1989	1640	1946	2326
Factor	121.4	142.9	157.9	125.5	145.9	155.3	124.2	147.8	172.0
% Change	--	+17.7	+30.1	--	+16.3	+23.7	--	+19.0	+38.5
TEA, ft-lb/ft^2	2.2	3.0	3.2	1.8	2.7	2.9	2.0	2.9	4.1
% Change	--	+36.4	+45.5	--	+50.0	+61.1	--	+45.0	+105.0
ZDT, psi	38.8	52.4	54.4	74.0	101.4	111.8	52.7	61.7	93.0
% Change	--	+35.1	+40.2	--	+37.0	+51.1	--	+17.1	+76.5
Brightness, %	14.4	17.9	23.6	22.2	26.6	28.8	16.6	20.2	33.4
% Change	--	+24.3	+63.9	--	+19.8	+20.7	--	+21.7	+101.2
Zero span factor, km	13.49	13.71	14.65	10.86	12.24	12.05	12.98	12.95	13.46
% Change	--	+1.6	+8.6	--	+12.7	+11.0	--	-0.2	+3.7

[a] Data obtained from Progress Report 1, Project 2697-53, entitled "Effect of Ozonation on Recycled Fiber", dated September 24, 1978

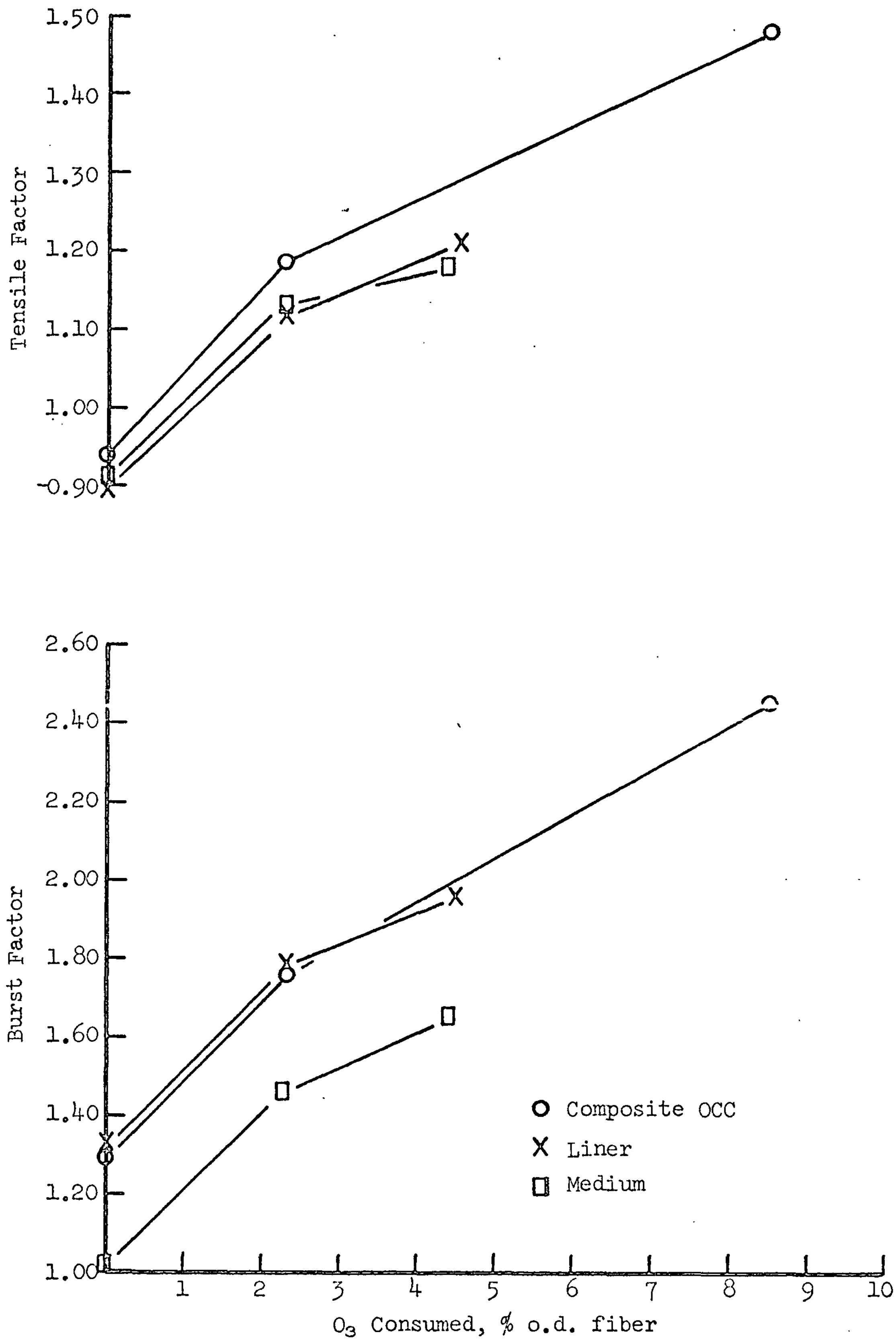

Figure 5. Burst and Tensile Factors on Ozonated Liner and Medium Fractions and Composite OCC

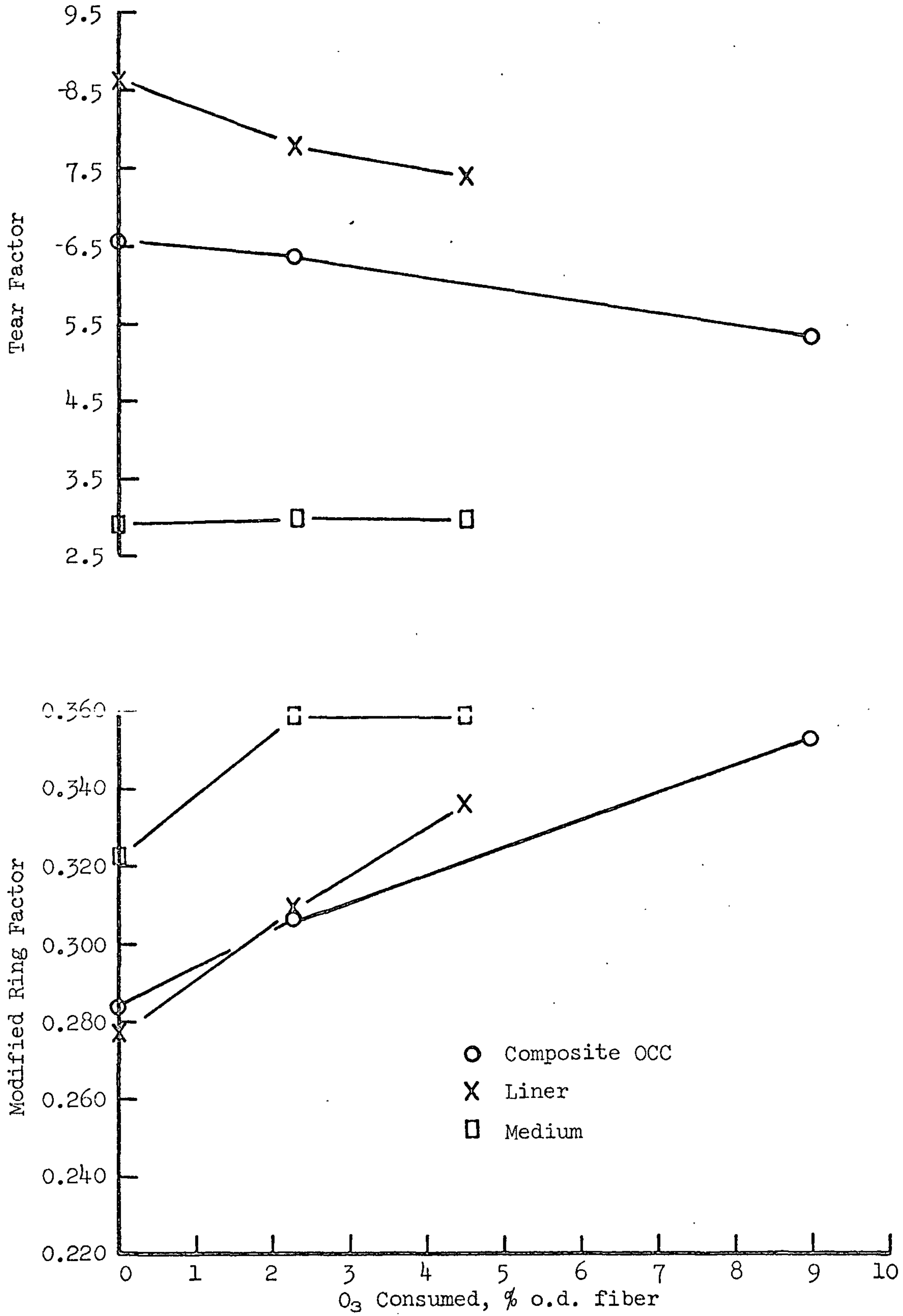

Figure 6. Tear and Modified Ring Results on Ozonated Liner and Medium Fractions and Composite OCC

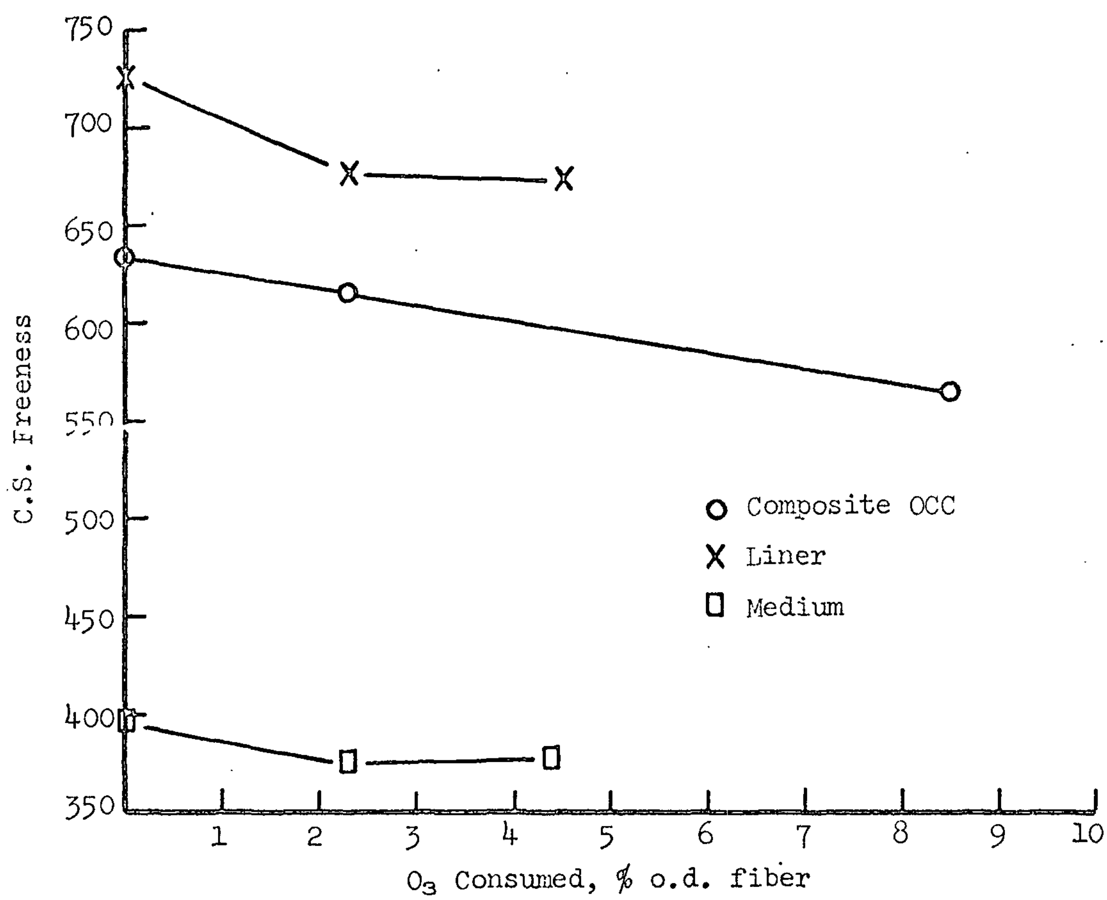

Figure 7. Freeness Results on Ozonated Liner and Medium Fractions and Composite OCC

softwood earlywood appears to respond to ozonation before the hardwood fibers and may serve to "explain" the differences in fiber surface responses. However, the fact that both the liner and the medium show similar percentage physical strength increases suggests that fiber modifications are taking place which are not visually evident.

Blending of Ozonated Liner and Medium Fractions

Studies have been initiated to blend the untreated and ozonated liner and medium fractions in various combinations to determine if trends in strength improvements will be modified. Initial experimental trials have been completed but analysis of the data is incomplete at this time.

Reduction of Ozone Application Rate

Early ozonation studies were carried out utilizing approximately 2% ozone in oxygen at a flow rate of 4 liters per minute. Physical test results on handsheets prepared from the ozonated fibers receiving less than 2.5% ozone consumed tended to be erratic. It was speculated that the erratic results were due to the laboratory process used in ozonation of the fibers which failed to allow sufficient time for diffusion of the O_3/O_2 gas through the fiber mass. Reaction times of 5 to 10 minutes corresponding to approximately 0.8 and 1.5% O_3 consumed were potentially insufficient to achieve necessary equilibrium. To check this hypothesis, the process was modified to provide a flow rate of 1 liter per minute of oxygen containing 2% ozone. Effective reaction time intervals were thus increased by a factor of four for a given ozone consumption level as shown in Figure 8.

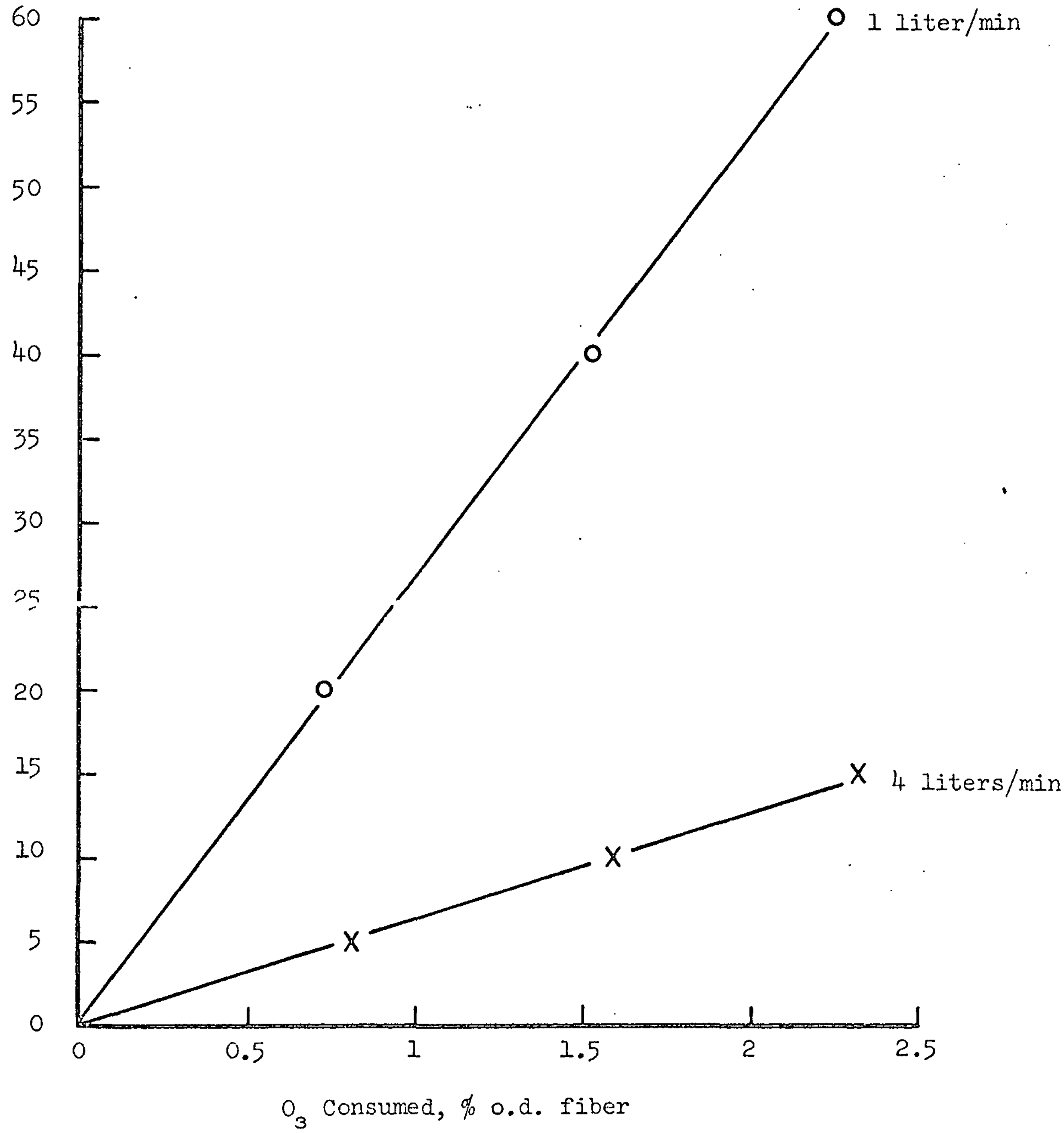

Figure 8. Effect of O_3/O_2 Addition Rate on O_3 Consumption and Treatment Time

Physical test results are shown in Table III. The changes in strength at the low O_3 levels were about the same at the two O_3 addition rates. However, most of the strength properties increased with increasing O_3 as expected.

C-stain and SEM studies of these fibers were undertaken. The C-stain studies suggested there may be a more uniform treatment of the fibers at the lower oxygen flow rate, but the results were not completely clear-cut. SEM studies proved to be more interesting. It was noted that trends in fiber modification could be observed between the successive increments of ozone consumption. Visual changes on the fiber surface at the 0.7% level were very limited and could not be readily characterized. At the 1.5% level, a trend toward "pitting or erosion" of the surface was definite, but little or no surface fibrillation could be observed. At the 2.3% level, "pitting or erosion" increased and the first evidences of limited fibrillation were observed. It is speculated that fibers undergo a series of modifications during ozonation, namely,

1. An initial molecular surface reaction which cannot be physically observed,

2. Secondly, a pitting or erosion of the apparently smooth fiber surface occurs which causes the surface to become "spongy" and reduces rigid surface stresses, and,

3. Finally, the surface becomes sufficiently modified to reveal surface microfibrils with reduced bonding to the parent fiber.

TABLE III

COMPARISON OF PROPERTIES OF HANDSHEETS PREPARED FROM OCC OZONATED AT DIFFERENT O_3 RATES

O_2 Feed Rate ($\sim$2% O_3 in O_2)	1 Standard liter/min				4 Standard liters/min[a]			
Ozonation time, min	0	20	40	60	0	5	10	15
Ozone applied, % of o.d. fiber	--	0.732	1.539	2.261	--	0.820	1.61	2.35
Ozone consumed, % of o.d. fiber	--	0.731	1.535	2.252	--	0.814	1.59	2.31
Reaction efficiency, %	--	99.9	99.7	99.6	--	99.2	98.5	98.3
C.S. freeness, cc	660	615	630	610	633	647	627	617
% Change	--	-6.8	-4.6	-7.6	--	+2.2	-0.9	-2.5
Basis weight, lb/M ft^2	13.5	13.5	13.6	13.6	13.2	13.9	13.6	13.2
Caliper, points	6.0	5.7	5.7	5.7	6.1	6.4	6.1	5.6
Apparent density	2.26	2.37	2.39	2.41	2.16	2.18	2.21	2.34
% Change	--	+4.9	+5.8	+6.6	--	+0.9	+2.3	+8.3
Bursting strength, psig	16.6	2.37	2.39	2.41	2.16	2.18	2.21	2.34
Factor	1.23	1.41	1.50	1.64	1.30	1.36	1.49	1.76
% Change	--	+14.6	+22.0	+33.3	--	+4.6	+14.6	+35.4
Mod. ring compression, lb/inch	4.0	3.6	4.3	4.3	3.8	4.3	4.2	4.0
Factor	0.300	0.267	0.312	0.319	0.284	0.311	0.312	0.307
% Change	--	-11.0	+4.0	+6.3	--	+9.5	+9.9	+8.1
Tear, grams	86.4	92.4	94.0	89.6	87.6	56.7	98.5	85.1
Factor	6.39	6.85	6.90	6.57	6.64	6.93	6.87	6.45
% Change	--	+7.2	+8.0	+2.8	--	+4.4	+3.5	-2.9
Tensile, lb/inch	12.0	12.9	13.7	14.7	12.4	13.3	13.8	15.6
Factor	0.89	0.96	1.00	1.08	0.94	0.95	1.01	1.19
% Change	--	+7.9	+12.9	+21.4	--	+1.1	+7.4	+26.6
Stretch, %	2.04	2.07	2.04	2.20	1.86	1.96	2.11	2.18
% Change	--	+1.5	0.0	+7.8	--	+5.4	+13.4	+17.2
Et, lb/inch	1640	1735	1866	1922	1640	1767	1802	1946
Factor	121.3	128.6	137.0	141.0	124.2	127.0	132.5	147.8
% Change	--	+6.0	+12.9	+16.2	--	+2.3	+6.7	+19.0
TEA, ft-lb/ft^2	2.2	2.4	2.4	2.8	2.0	2.2	2.5	2.9
% Change	--	+9.1	+9.1	+27.3	--	+10.0	+25.0	+45.0
ZDT, psi	45.2	52.4	53.4	57.6	52.7	45.4	51.1	61.7
% Change	--	+15.9	+18.1	+27.4	--	-13.9	-3.0	+17.1
Brightness, %	16.4	17.1	19.0	20.2	16.6	18.2	19.1	20.2
% Change	--	+4.3	+15.9	+23.2	--	+9.6	+15.1	+21.7
Zero span factor, km	13.25	12.86	13.98	13.42	12.98	12.94	12.81	12.95
% Change	--	-2.9	+5.5	+1.3	--	-0.3	-1.3	-0.2

[a]Data obtained from Progress Report 1, Project 2697-53, entitled "Effect of Ozonation on Recycled Fiber", dated September 24, 1978

Since a general trend toward strength increase is shown even at the 0.7% ozone level, it may be suggested that all of the above changes affect one or more of the strength properties; but the rate at which a given property is increased may be related to a certain critical level of modification of the fiber surface. Early changes might be expected to modify fiber stiffness or conformability, but real increases in fiber surface may not be achieved until microfibrillation occurs. It appears that more fundamental studies are required to better understand ozone/fiber reactions. Current studies have suggested hypotheses for the reaction mechanism but, in reality, have raised additional questions that require answers before ozone/fiber reaction conditions can be fully defined and optimized.

Rapid Ozonation of Fiber Pads

Several screening trials were attempted to determine if ozonation would proceed rapidly in an excess of ozone. To achieve these goals, it was necessary to ozonate only small quantities of fiber in an appropriate reaction unit. Thin fiber pads of approximate 1-1/4 inch diameters were prepared in a Gelman filter unit at a consistency of 30-50%. Consistency was difficult to maintain but appeared to approximate consistencies normally achieved by fluffing. Pads contained about 35 mg of o.d. fiber. The pad was placed into an aluminum Gelman in-line filter which became the reaction vessel. Oxygen containing 2% ozone was applied to the pad at a flow rate of 4 liters per minute. Pads were treated for 1, 2, 4, and 8 minutes. Fibers were stained with C-stain. Color changes were compared with color changes occurring at known percentages of ozone consumed. Color changes on fibers from the 1 minute ozonation interval suggested the fibers consumed in excess of 2.3% ozone. At the 8 minute ozonation interval, color changes were roughly equivalent to 12% ozone consumed. Comparable time intervals under previous ozonation procedures were 15 to 90 minutes, respectively. The preliminary trials suggest that ozonation proceeds rapidly when sufficient ozone is present and processing conditions are optimized. Results indicate reaction rate was increased by a factor of ten. A continuous process based upon a moving web may be feasible.

Consistency Studies

The literature reports consistency of the pulp is critical to effective ozonation. Consistencies above 30% are reported to be necessary to provide reasonable reaction efficiencies. A study has been initiated to determine the desirable consistency range for OCC pulp. Trials in process cover pulp consistencies from 30 to 60%.

Patent Review

A preliminary patent review is being prepared covering the ozonation process and equipment. It will also assist equipment design and planning for pilot scale operations should laboratory trials continue to be favorable.

Future Work

Current in process studies will be completed. They include:

1. Determination of the ozonation characteristics of commercial OCC from several sources.

2. The effect of blending ozonated liner and medium fractions on strength.

3. Determine the effects of pulp consistency on the ozonation process.

In addition, studies covering the following are being planned:

1. Effects of temperature variations on the ozonation process.

2. Determination of the nature and amount of possible soluble products resulting from ozonation.

3. Study of the strength properties of ozonated OCC/virgin pulp blends.

4. Determination of the effects of pre- and post-refining on ozonated pulps.

IV. Chemical Treatments

A series of trials are underway to compare the effectiveness of various chemical treatments of recycled OCC with each other and with ozonation. The treatments under study and initial results obtained are are briefly summarized below.

Caustic Treatments -- Atmospheric Pressure

Repulped OCC at 4% consistency was treated with NaOH using concentrations of 3 and 5% based on the weight of ovendry (o.d.) fiber. These concentrations were selected based on the work of Seifert and Long* who reported that an increase of about 25% in bursting strength resulted using 4% NaOH. Two temperatures were employed for this study, namely, room temperature (77°F) and about 198°F. The stocks were treated for two hours at each concentration and temperature level. Beating curves were obtained at each condition, as well as on the untreated control.

The yields for these caustic treatments ranged between about 94-96%. These yields are about the same as obtained by Seifert and Long.

The results obtained using the 3 and 5% caustic treatments at 198°F are summarized in Table IV. Figure 9 shows that the caustic treatments decreased the freeness by about 50 cc at each beating interval which is probably due to swelling of the fibers by the caustic. Figure 10 shows that the caustic treatments generally increased the burst and tensile factors at each beating interval. The percentage improvements in these

*Seifert, P. and Long, K. J., Tappi 57, no. 10: 69-72 (October 1974).

TABLE IV

EFFECT OF CAUSTIC TREATMENTS AT ATMOSPHERIC PRESSURE
(198°F TREATMENT TEMPERATURE)

Treatment, % NaOH at 198°F	C.S. Freeness, cc	Basis Weight, lb/M ft²	Caliper, mil	Apparent Density		Burst Factor		Tensile Factor	
				lb/mil	Diff. %	psi/lb	Diff. %	lb/in/lb	Diff. %
0 Minute Beating Time									
0	665	13.8	6.1	2.25	---	1.28	---	0.89	---
3	615	13.6	5.3	2.56	+13.8	1.49	+16.4	1.03	+15.7
5	625	13.5	5.1	2.64	+17.3	1.62	+26.6	1.12	+25.8
5 Minute Beating Time									
0	605	13.6	5.8	2.36	---	1.66	---	1.18	---
3	555	13.6	5.0	2.74	+16.1	1.96	+18.1	1.26	+6.8
5	540	13.9	5.1	2.75	+16.5	2.02	+21.6	1.30	+10.2
10 Minute Beating Time									
0	545	13.8	5.5	2.52	---	1.94	---	1.34	---
3	490	13.7	4.8	2.84	+12.7	2.21	+13.9	1.42	+6.0
5	480	13.6	4.7	2.91	+15.5	2.34	+20.6	1.52	+13.4
20 Minute Beating Time									
0	375	14.0	5.0	2.78	---	2.62	---	1.54	---
3	315	13.4	4.5	2.93	+5.4	2.60	-0.8	1.73	+12.3
5	330	13.7	4.5	3.05	+9.7	2.57	-1.9	1.71	+11.0
30 Minute Beating Time									
0	225	13.0	4.6	2.79	---	2.90	---	1.76	---
3	195	13.4	4.4	3.04	+9.0	2.92	+0.7	1.81	+2.8
5	190	13.5	4.3	3.12	+11.8	3.12	+7.6	1.86	+5.7

Note: Percent differences are based on the control results at each beating time.

TABLE IV (Continued)

EFFECT OF CAUSTIC TREATMENTS AT ATMOSPHERIC PRESSURE
(198°F TREATMENT TEMPERATURE)

Treatment, % NaOH at 198°F	Tear Factor g/lb	Tear Factor Diff. %	Modified Ring Factor lb/in/lb	Modified Ring Factor Diff. %	Et Factor lb/in/lb	Et Factor Diff. %	TEA ft-lb/ft^2	TEA Diff. %	Stretch, %
				0 Minute Beating Time					
0	6.21	---	0.304	---	125.3	---	1.8	---	1.73
3	6.43	+3.5	0.334	+9.9	125.8	+0.4	2.4	+33.3	2.03
5	6.45	+3.9	0.281	-7.6	135.5	+7.3	2.9	+61.1	2.25
				5 Minute Beating Time					
0	5.94	---	0.331	---	143.2	---	2.8	---	2.09
3	6.42	+8.1	0.337	+1.8	143.5	-3.2	2.9	+3.6	2.08
5	6.17	+3.9	0.301	-9.1	143.2	-3.4	3.3	+17.9	2.24
				10 Minute Beating Time					
0	5.85	---	0.377	---	152.1	---	3.5	---	2.14
3	6.03	+3.1	0.340	-9.8	152.5	+0.3	3.6	+2.9	2.28
5	5.51	-5.8	0.353	-6.4	160.9	+5.8	3.9	+11.4	2.32
				20 Minute Beating Time					
0	5.34	---	0.393	---	163.5	---	4.6	---	2.51
3	5.03	-5.8	0.352	-10.4	171.7	+5.0	4.9	+6.5	2.59
5	5.33	-0.2	0.375	-4.6	174.0	+6.4	4.5	-2.2	2.36
				30 Minute Beating Time					
0	5.02	---	0.415	---	182.8	---	4.9	---	2.56
3	5.11	+1.8	0.355	-16.9	173.9	-2.1	5.1	+4.1	2.56
5	4.89	-2.6	0.374	-9.9	185.6	+1.5	5.5	+12.2	2.67

Note: Percent differences are based on the control results at each beating time.

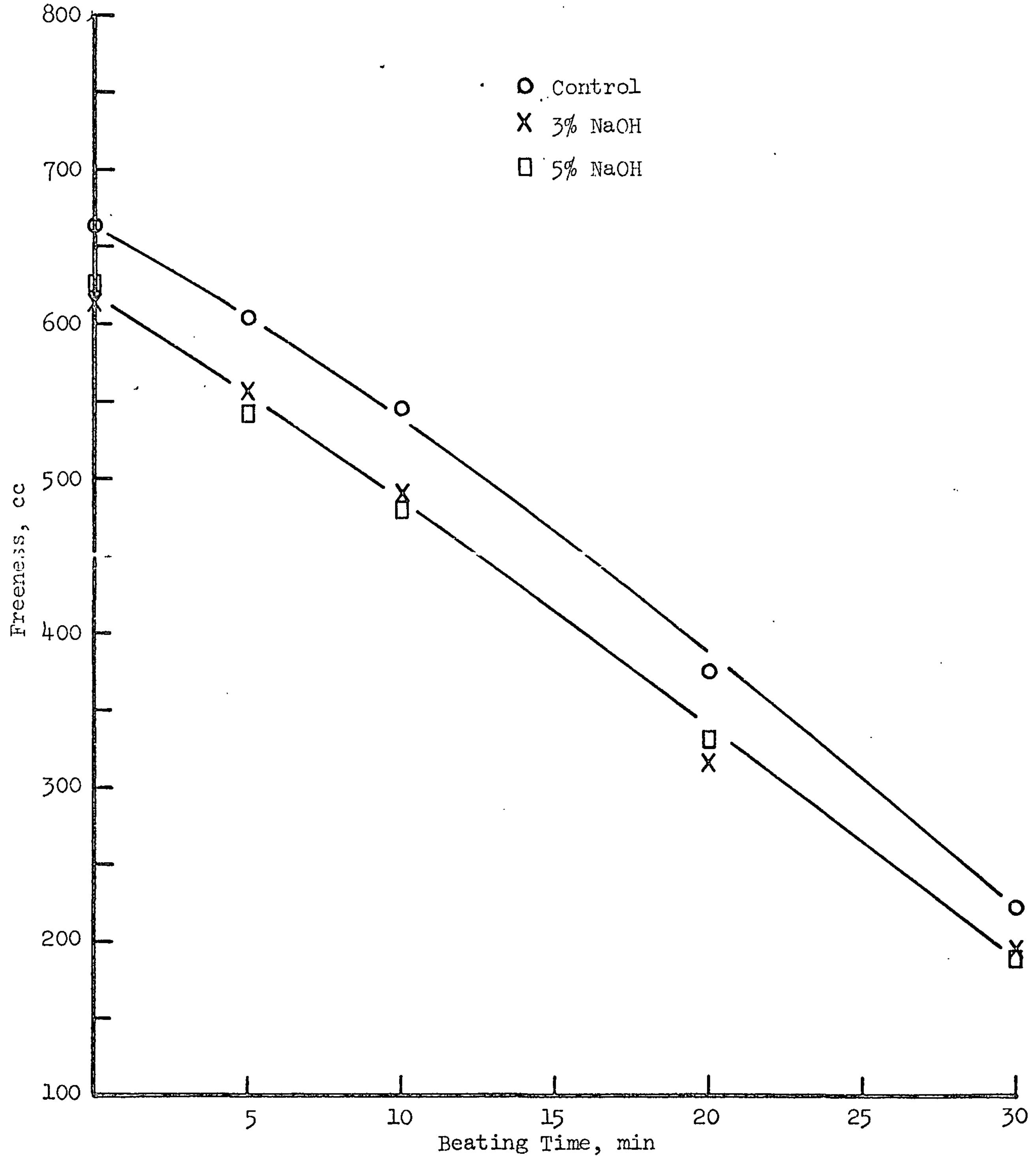

Figure 9. Effect of Caustic Treatments at Atmospheric Pressure on Freeness

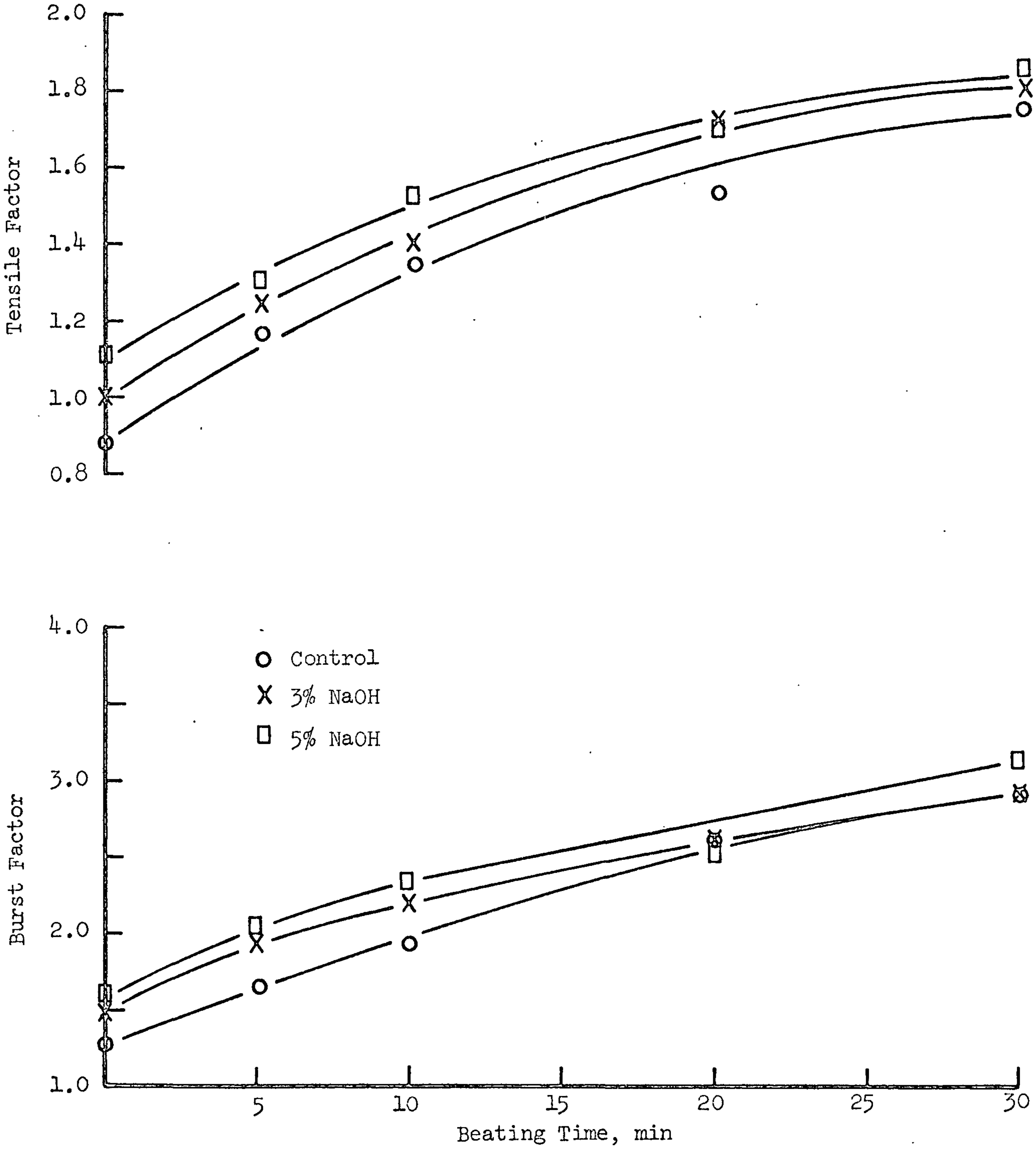

Figure 10. Effect of Caustic Treatments at Atmospheric Pressure on Burst and Tensile

properties tended to decrease as beating progressed. It appears that the improvements in burst and tensile at the 0 beating time were approximately proportional to the caustic concentration (see Figure 11). Past work on ozonation indicated that a 35% improvement in burst could be achieved with 2.3% O_3 consumption. The results in Figure 11 suggest that caustic treatments near 7-8% might be required to achieve a 35% burst improvement. On the other hand, the tensile improvement of 25.8% with 5% caustic was approximately the same as that obtained at the 2.3% O_3 consumption level (27%).

The tear factor results in Figure 12 show that the caustic treatments tended to slightly increase the tearing strength at the shorter beating times. However, the differences are probably not significant. The effects of the caustic treatment on modified ring compression in Figure 12 are somewhat erratic which is probably due, in part, to the difficulties of carrying out compression tests on thin, light-weight sheets. With this reservation, the results suggest the caustic treatments tended to lower the modified ring strength in most cases at the various beating intervals.

When the burst and tensile factors are plotted against freeness, the results show that the control and caustic treated samples exhibit about the same test level at a given freeness level (Figure 13). This indicates that the increases in tensile and burst strength achieved with these caustic treatment conditions are about the same as would be obtained by mechanical refining.

The above trials were all carried out at low consistency. A limited trial is also in progress involving a high consistency caustic treatment of OCC. It has been speculated that such a treatment might have advantages

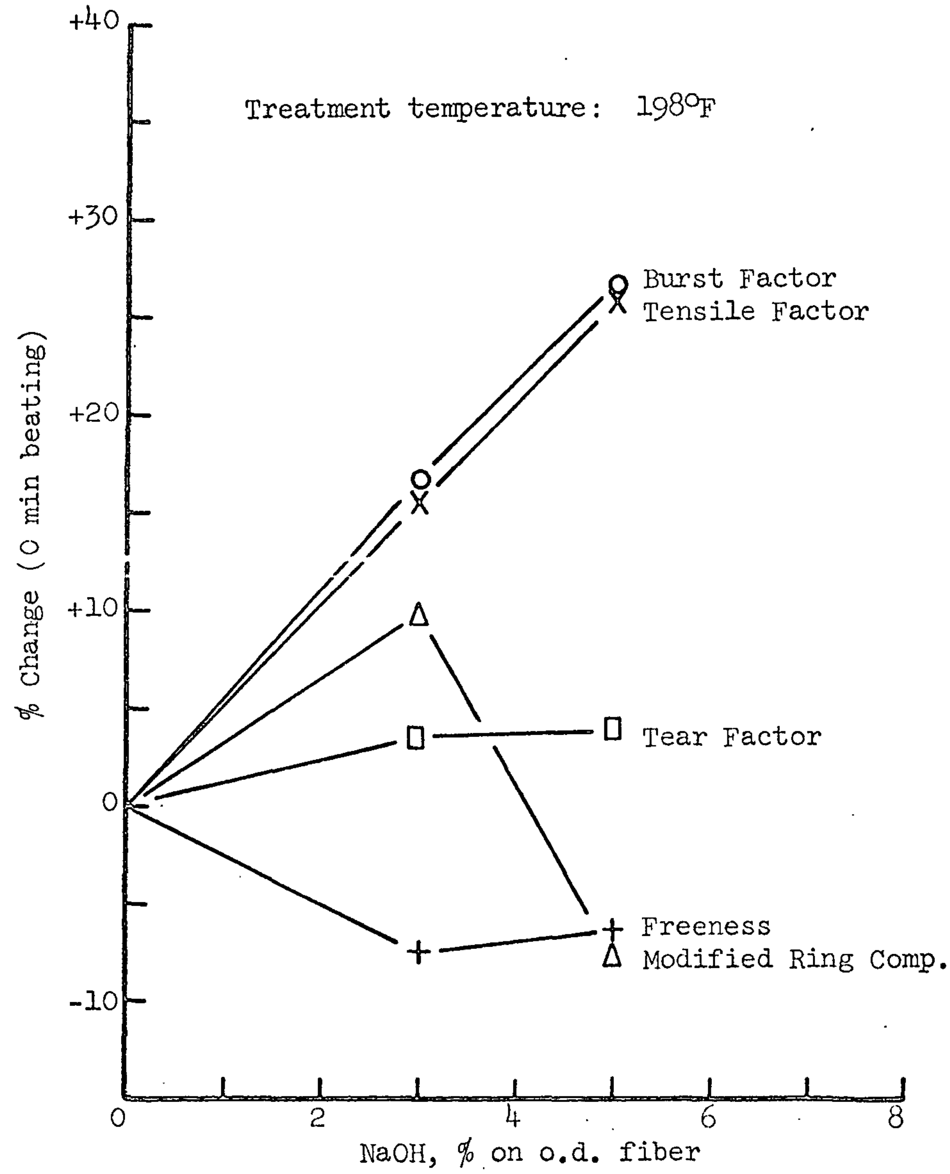

Figure 11. Effect of Caustic Treatment Concentration on Various Sheet Properties

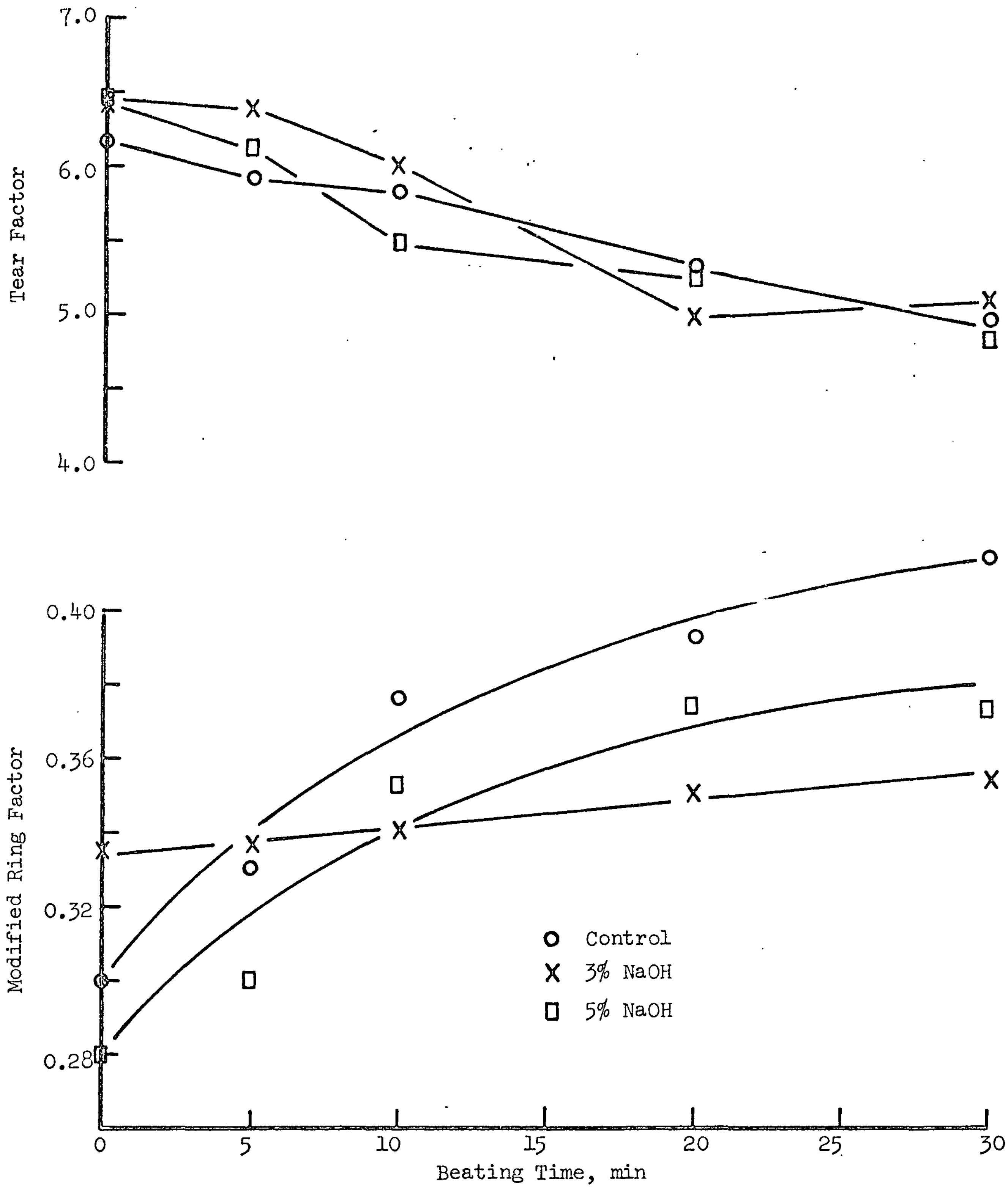

Figure 12. Effect of Caustic Treatments at Atmospheric Pressure on Tear and Modified Ring Compression

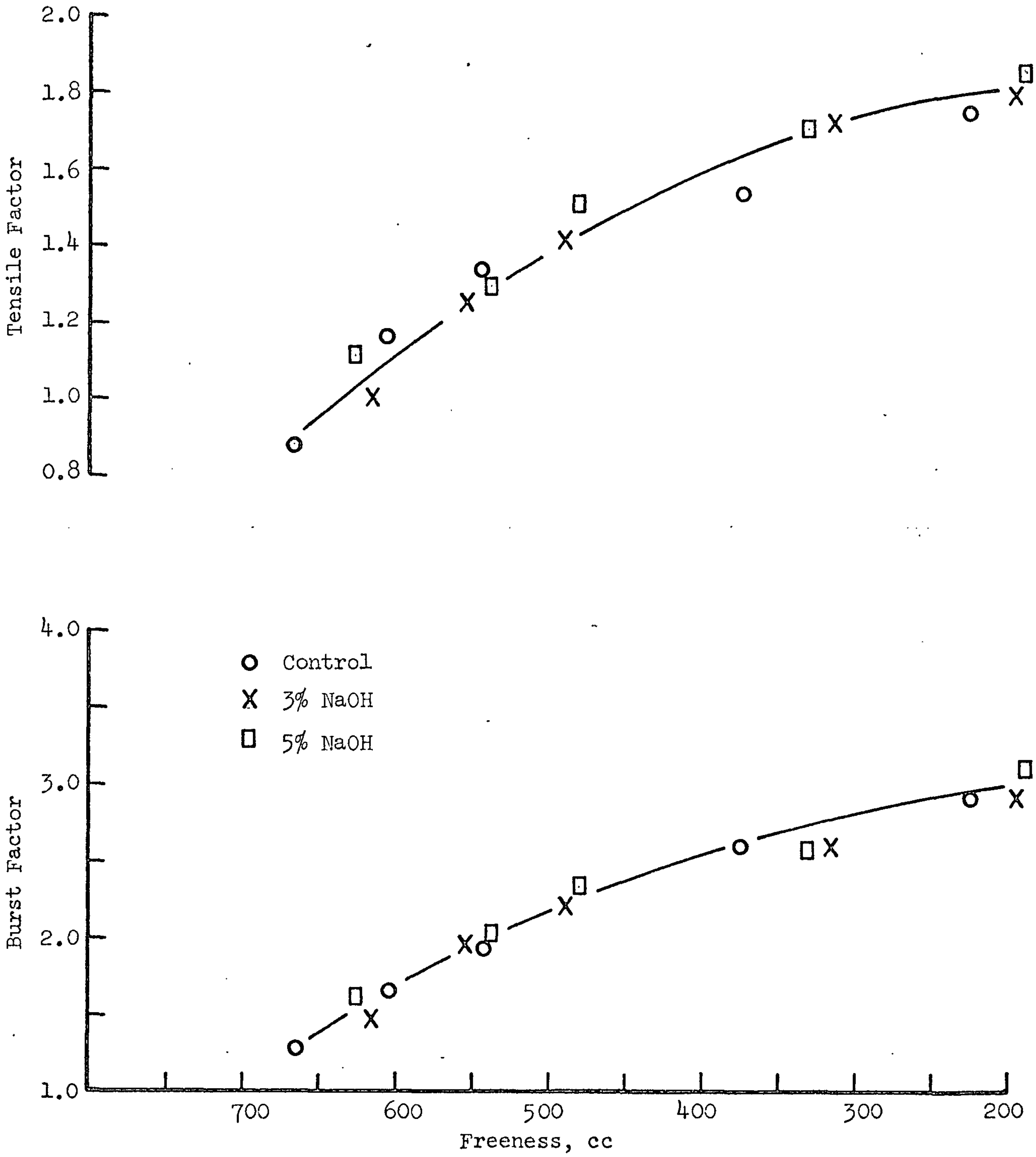

Figure 13. Tensile and Burst vs. Freeness on Caustic Treated OCC

for treating shredded OCC which is moistened to the desired consistency level. After treatment the stock would be diluted for screening and cleaning.

Pressurized chemical treatments

The following treatments are being carried out in the Asplund defibrator to simulate the effect of chemical treatments in A/D systems. The processing has been carried out using 135 grams o.d. fiber with the selected amount of chemical. The consistency of the stock was about 25-30% with a dwell time in the Asplund preheater of 5 minutes with 100 psi steam. The following chemical levels have been used.

1. Caustic soda (NaOH): 3 and 5% on o.d. fiber
2. Sodium carbonate: 8 and 13.25%
3. Hydrogen peroxide (1%) in an alkaline slurry

In addition to the above, a mild oxygen treatment to give a moderate reduction in lignin content has been carried out. For this purpose, repulped OCC at about 32% consistency was treated with 90 psi O_2 at 110°C for 20 minutes. A small amount of magnesium ion was also added to prevent attacking the cellulose.

The above "cooks" have all been completed and the handsheet preparation at each beating interval is in process.

V. Chemical Additives

For comparison with the chemical treatments, including ozonation, a series of trials will be initiated using selected bonding and drainage aids to achieve acceptable strength and water-removal properties. These trials were deferred in order to carry out the chemical treatments; however, work on this phase will be started in early 1979. The initial materials are on hand and will include cationic corn starch, cationic potato starch, guar or locust beam gum and synthetic drainage aids.

VI. OCC and Virgin Primary Properties at "Standard" Sheet Weight

In the ozonation and chemical treatment studies, the amounts of stock which could be conveniently processed were limited. Therefore, for strength comparison purposes, the handsheets were prepared using the standard handsheet weight (approximately 60 g/m^2 or 13 $lb/M\ ft^2$). Because portions of the initial work on this study were carried out using a heavier sheet weight (ca. 42 $lb/M\ ft^2$), it was desirable to obtain new beater curves on the OCC and virgin primary stocks to compare the effectiveness of the various treatments and additives. In addition, data were obtained at both 50 and 90 psi wet pressing levels at selected beater intervals in order to permit future comparisons of chemical treatments effectiveness with increased wet pressing.

The handsheet results on the OCC are shown in Tables V and VI for the 50 and 90 psi wet pressing pressures, respectively. Tables VII and VIII contain the corresponding results on the virgin primary.

Figures 14 and 15 compare the OCC and virgin kraft properties at the standard (50 psi) wet pressing pressure. As may be noted in Figure 14, the burst factor of the OCC is about 39% lower than the virgin kraft at an arbitrarily selected freeness level of 600 cc. This is about the degree of improvement obtained in past work with ozone treatments at the 2.3% O_3 consumption level. It is somewhat greater than was achieved with the 5% caustic treatment discussed previously.

Figures 16 (virgin kraft) and 17 (OCC) show that the burst and tensile factors were slightly higher at the 90 psi wet pressing pressure as would be expected.

TABLE V

OCC HANDSHEET PROPERTIES AT 50 PSI WET PRESSING PRESSURE

Physical Property	Beater Interval 0 min	5 min	10 min	20 min	30 min
C.S. freeness, cc	665	605	545	375	225
Basis weight, lb/1000 sq. ft	13.8	13.6	13.8	14.0	13.0
Thickness, mils	6.1	5.8	5.5	5.0	4.6
Apparent density, lb/1000 sq. ft-mil	2.25	2.36	2.52	2.78	2.79
Burst, psig	17.6	22.5	26.8	36.8	37.6
Factor	1.28	1.66	1.94	2.62	2.90
Modified ring, lb/inch	4.2	4.5	5.2	5.5	5.4
Factor	0.304	0.331	0.377	0.393	0.415
Tensile, lb/inch	12.3	16.1	18.6	21.6	22.8
Factor	0.89	1.18	1.34	1.54	1.76
Et, lb/inch	1741	2016	2101	2290	2374
Factor	126.3	148.2	152.1	163.5	182.8
Tear, grams	85.6	80.8	80.8	74.8	65.2
Factor	6.21	5.94	5.85	5.34	5.02
Stretch, %	1.73	2.09	2.14	2.51	2.56
TEA, ft-lb/sq. ft	1.8	2.8	3.5	4.6	4.9

TABLE VI

OCC HANDSHEET PROPERTIES AT 90 PSI WET PRESSING PRESSURE

Physical Property	Beater Interval				
	0 min	5 min	10 min	20 min	30 min
C.S. freeness, cc	665	605	545	375	225
Basis weight, lb/1000 sq. ft	13.8	13.9	13.8	13.7	13.8
Thickness, mils	5.9	5.4	5.1	4.8	4.6
Apparent density, lb/1000 sq. ft-mil	2.35	2.58	2.70	2.85	3.01
Burst, psig	18.0	24.5	29.4	35.2	39.9
Factor	1.30	1.76	2.13	2.57	2.90
Modified ring, lb/inch	4.0	4.6	4.8	5.4	5.5
Factor	0.290	0.331	0.348	0.394	0.399
Tensile, lb/inch	13.6	17.4	19.2	22.6	24.2
Factor	0.98	1.25	1.39	1.66	1.76
Et, lb/inch	1854	2115	2182	2318	2522
Factor	132.5	151.9	158.0	169.7	183.1
Tear, grams	87.6	82.0	77.6	67.6	60.4
Factor	6.33	5.89	5.62	4.95	4.39
Stretch, %	1.85	2.06	2.09	2.73	2.60
TEA, ft-lb/sq. ft	2.1	3.0	3.3	5.2	5.3

TABLE VII

VIRGIN PRIMARY HANDSHEET PROPERTIES AT 50 PSI WET PRESSING PRESSURE

Physical Property	Beater Interval 5 min	20 min	35 min	50 min	60 min
C.S. freeness, cc	765	725	640	435	270
Basis weight, lb/1000 sq. ft	13.6	14.4	13.8	13.6	14.1
Thickness, mils	6.2	5.6	5.1	4.6	4.5
Apparent density, lb/1000 sq. ft-mil	2.20	2.55	2.72	2.93	3.12
Burst, psig	18.1	31.7	37.1	42.4	47.8
Factor	1.33	2.20	2.69	3.12	3.40
Modified ring, lb/inch	4.4	5.2	5.0	5.4	5.8
Factor	0.324	0.361	0.362	0.397	0.409
Tensile, lb/inch	13.3	19.3	21.7	25.1	28.6
Factor	0.98	1.34	1.58	1.85	2.03
Et, lb/inch	1868	2106	2246	2422	2765
Factor	137.2	146.5	163.0	178.1	196.7
Tear, grams	168.8	127.2	107.2	87.2	76.4
Factor	12.39	8.85	7.78	6.41	5.43
Stretch, %	1.43	2.00	2.48	2.73	2.47
TEA, ft-lb/sq. ft	1.5	3.1	4.6	5.8	5.7

TABLE VIII

VIRGIN PRIMARY HANDSHEET PROPERTIES AT 90 PSI WET PRESSING PRESSURE

Physical Property	Beater Interval 20 min	35 min	50 min
C.S. freeness, cc	725	640	435
Basis weight, lb/1000 sq. ft	14.2	14.4	13.2
Thickness, mils	5.0	4.9	4.5
Apparent density, lb/1000 sq. ft-mil	2.80	2.97	2.97
Burst, psig	33.4	43.2	44.1
Factor	2.36	2.99	3.33
Modified ring, lb/inch	4.8	5.3	5.2
Factor	0.338	0.368	0.394
Tensile, lb/inch	21.5	24.6	25.9
Factor	1.52	1.70	1.96
Et, lb/inch	2264	2502	2756
Factor	159.9	173.2	208.2
Tear, grams	116.0	94.8	79.2
Factor	8.19	6.56	5.98
Stretch, %	2.23	2.42	2.73
TEA, ft-lb/sq. ft	4.0	5.0	5.3

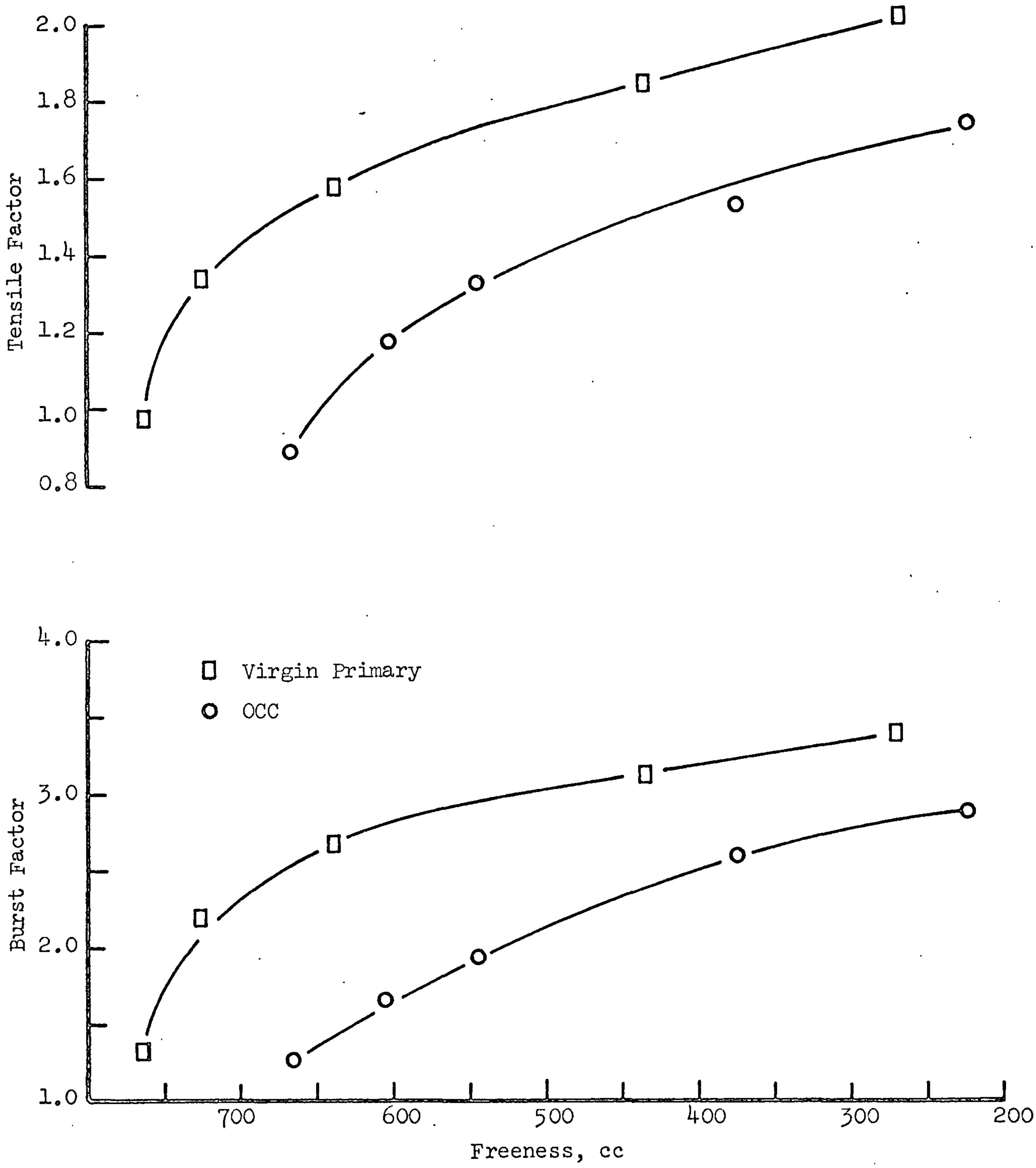

Figure 14. Tensile and Burst Properties on OCC and Virgin Kraft (50 psi wet pressing)

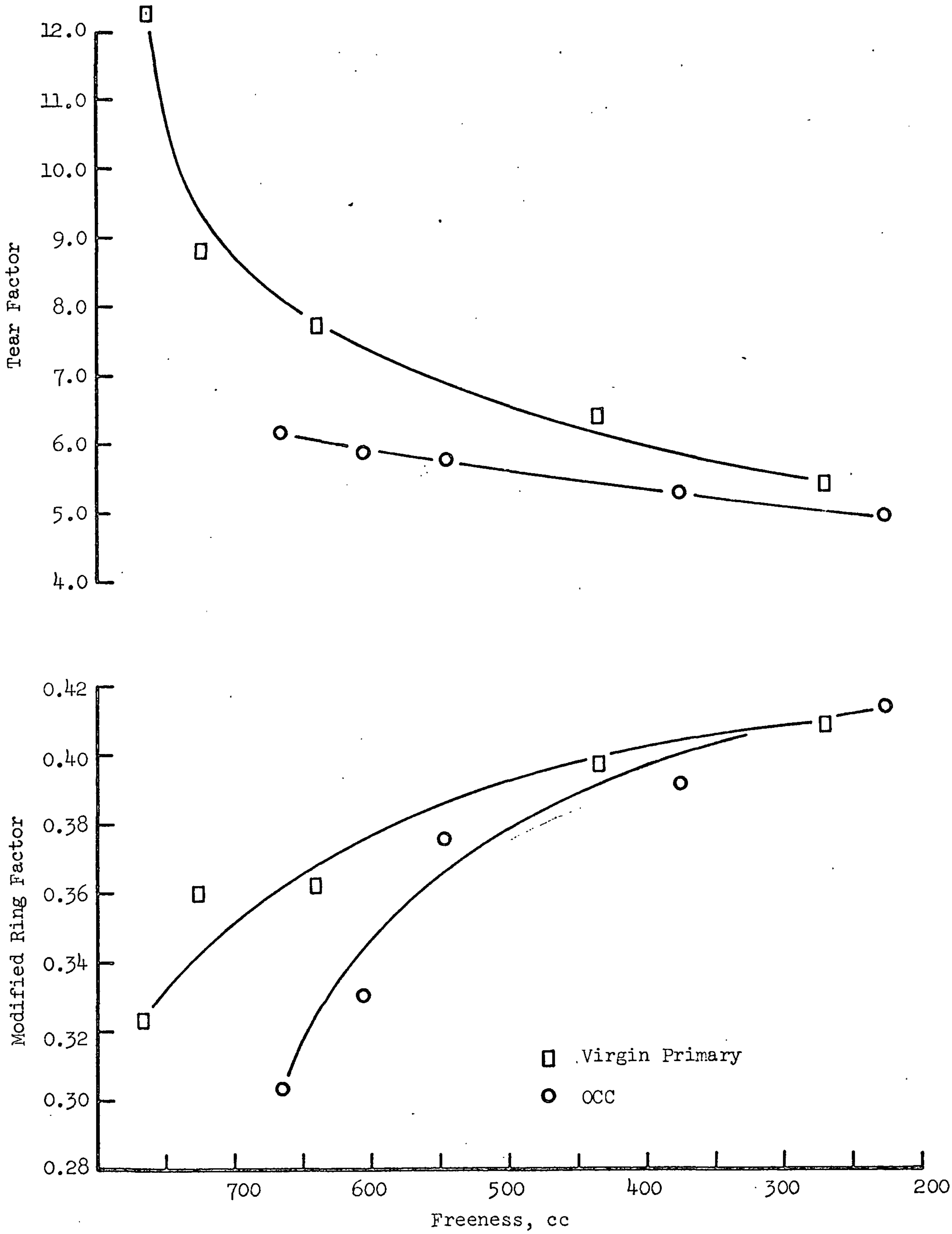

Figure 15. Tear and Modified Ring Compression Factors on OCC and Virgin Kraft (50 psi wet pressing)

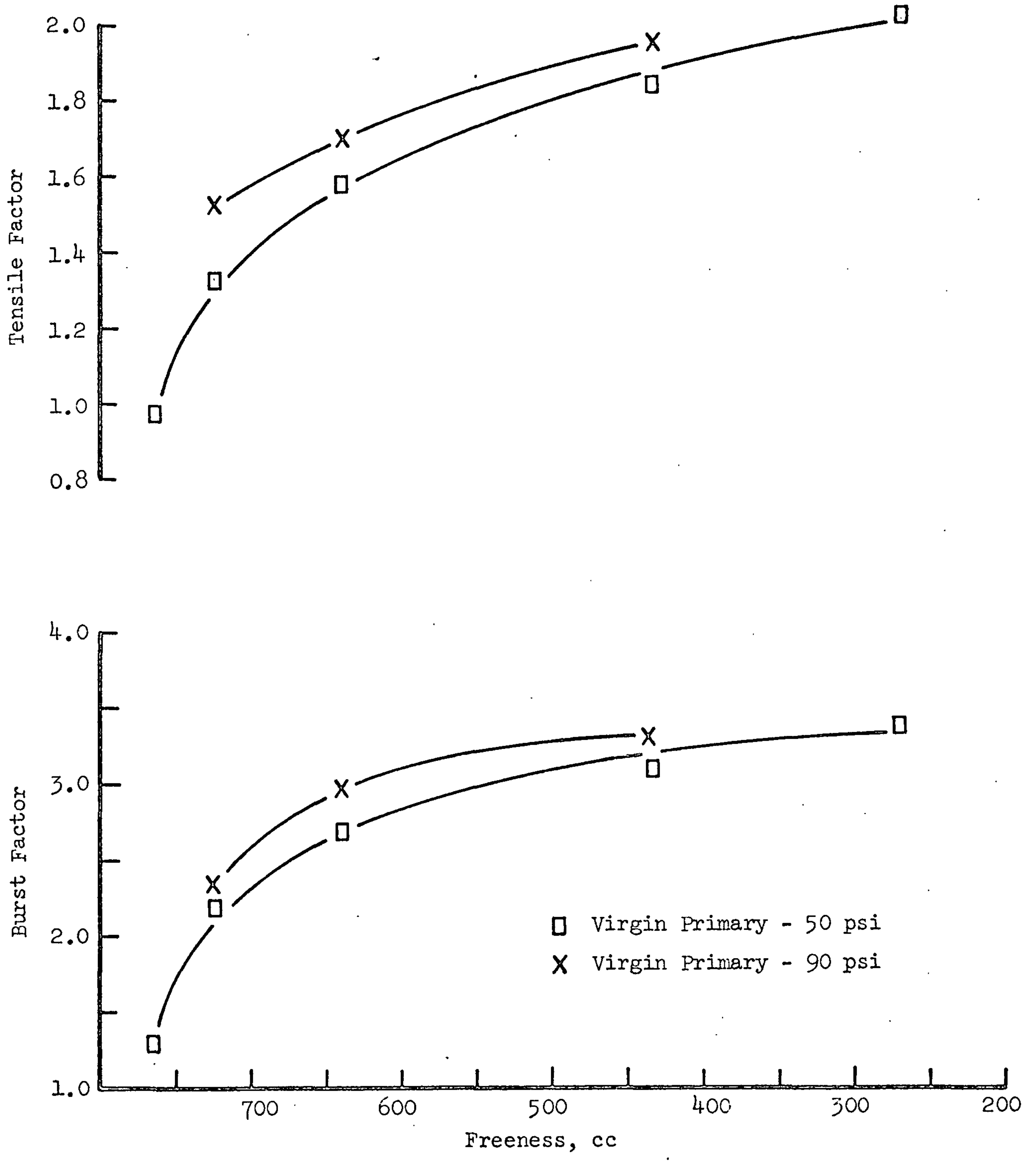

Figure 16. Effect of Wet Pressing on Virgin Kraft Burst and Tensile Factors

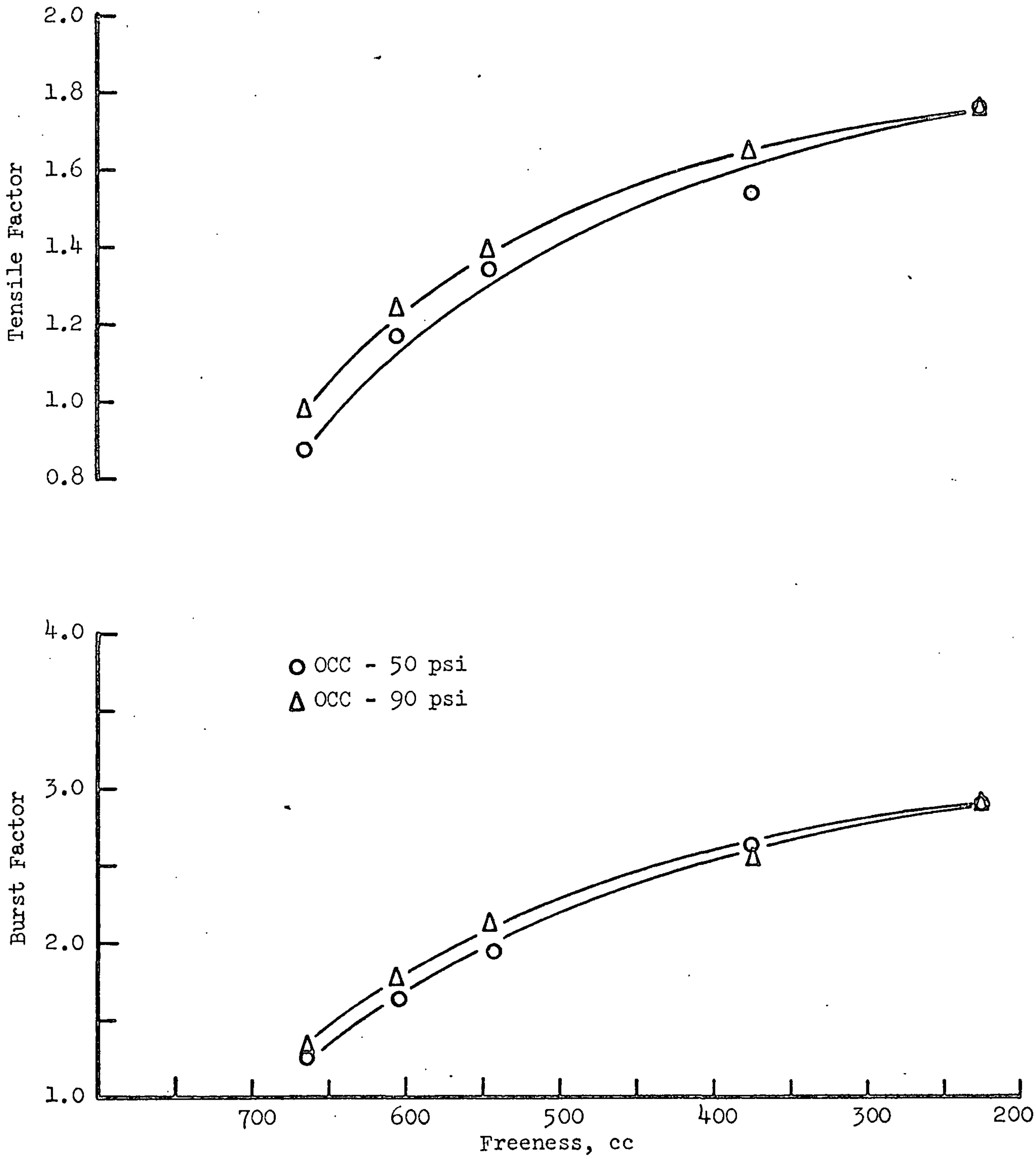

Figure 17. Effect of Wet Pressing on OCC Burst and Tensile Factors